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Effect of Silicon Carbide on Devitrification of a Glass Coating for Reusable Surface Insulation

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National Aeronautics
and Space Administration

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SUMMARY

Devitrification (nucleation and growth of cristobalite) has been investigated in the LI-0042 coating used for the Space Shuttle surface insulation. Supporting evidence for mechanisms of devitrification in this coating is presented and discussed. The LI-0042 coating formulation was prepared both with and without silicon carbide (SiC) and was exposed to simulated reentry thermal cycles. The coating with SiC experienced much higher rates of devitrification than the coating without SiC and produced 3 to 5 times more cristobalite than the coating without SiC. A comparison of thermal exposures in air with exposures in a vacuum of coating that contained SiC showed that significant cristobalite formation occurred only when oxidation of the SiC was possible. The effect of exposing the coating to water before each thermal cycle was also investigated.

INTRODUCTION

The reusable surface insulation (RSI) which will thermally protect the Space Shuttle during reentry is a low-density (144 kg/m^3), rigidized fibrous silica material. The requirements of high-temperature operation, moisture resistance, and high emittance dictate a need for the RSI surface to be protected with a suitable continuous coating. One of several coatings which was developed to satisfy this need was the LI-0042. This two-layered coating has an outer layer of borosilicate glass which contains a dispersion of silicon carbide (SiC) particles to increase surface emittance. The base layer is high-purity silica glass which is used to seal the surface of the RSI.

Environmental and thermal testing of the LI-0042 revealed that it could not survive for more than about 50 simulated Shuttle mission cycles without cracking, spalling, and losing waterproof qualities (ref. 1). An evaluation of coatings that failed during these tests showed that large amounts of cristobalite (a crystalline polymorph of silica) were present. In no case was cristobalite detectable at the beginning of tests. Cristobalite in a silica glass which is exposed to cyclic heating is destructive because of the large volume changes associated with the transformation of the cristobalite from the high-temperature (β) to the low-temperature (α) form.

The LI-0042 was designed with the expectation that negligible devitrification (nucleation and growth of cristobalite) would occur under the thermal conditions imposed during Space Shuttle service. Therefore, the extent of devitrification observed in the tests of reference 1 was unexpected. Much of the devitrification was attributed to contamination with water and sea salts from the Space Shuttle launch-pad environment; however, significant devitrification also occurred in coating specimens which were exposed only to thermal cycles. The present investigation was therefore undertaken to identify causes and mechanisms of devitrification in the LI-0042 coating. The effects of SiC were studied in detail because it has the potential of causing devitrification

in glass by oxidation and/or heterogeneous nucleation. Evidence of these mechanisms is presented and discussed. Since water can also play an important role in devitrification, its effects were investigated and are discussed as well.

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MATERIALS AND SPECIMENS

Six specimens of RSI were prepared for coating from uncoated LI-0900. The specimens were finished to final dimensions of 32 mm by 32 mm by 25 mm (± 2 mm) by dry grinding with No. 180 grit SiC paper. Dust was removed with a stream of compressed (138 kPa) filtered air. The specimens were then subjected to a thermal soak for 12 hr at 810 K and 101.3 kPa to remove an organic hydrophobic agent which was in the originally supplied RSI.

The six specimens were divided into 2 groups for application of coating slurries. Slurry A was prepared according to the process specification for LI-0042 given in the appendix, except that the SiC powder used was No. 600 grit rather than No. 1200 grit. Slurry B was prepared according to specification except that SiC was omitted. Specimens 1, 2, and 3 were coated with slurry A and specimens 4, 5, and 6 with slurry B. Each specimen was coated on five sides in accordance with specification except that the slurry was hand brushed. The specimens were fired in accordance with the specification. One specimen of each group (specimens 3 and 6) was prepared with a thermocouple (type R) embedded in the coating, as illustrated in figure 1. These two specimens were used for temperature control during thermal tests. A seventh specimen, 32 mm by 32 mm by 25 mm, was cut from the corner of a LI-0900 RSI tile coated with LI-0042, as supplied by Lockheed Missiles and Space Company, Inc. (LMSC). This specimen was therefore coated on three sides. Specimen information is compiled in table I.

TEST PROCEDURES

Thermal Testing

Testing at atmospheric pressure.— Before testing, specimens 1, 2, 4, and 5 were inspected, photographed, and measured. Measurements consisted of determining the height at the center of the test face (face opposite uncoated side) and at the edges. A dial gage was used to make measurements to the nearest 0.025 mm. This procedure was repeated after testing. The control specimens (specimens 3 and 6) were not measured.

Specimens 1, 2, 4, and 5 were each exposed to 100 cycles of the thermal profile shown in figure 2. This profile approximates a standard profile used to simulate reentry thermal exposures of high-temperature RSI (HRSI). One

specimen of each coating (specimens 2 and 5) was exposed to distilled deionized water (resistance ≥ 0.8 megohm) before each thermal cycle. (Water exposure consisted of floating a specimen with the test face down for 10 sec.) After 5 cycles, however, the exposure time was reduced to 2 sec for specimen 2 (coating A) because of very rapid water absorption. (The specimen would begin to sink in 10 sec.) After water exposure, specimens were freed of standing water with compressed air and immediately exposed to the next thermal cycle.

The heat source used for thermal exposures was a radiant heater which is described in reference 1. The heater was controlled with the feedback signal from the thermocouple in a control specimen (specimen 3 (coating A) or specimen 6 (coating B)). Each specimen (specimens 1, 2, 4, and 5) was tested separately. The surface temperature in each case was assumed to be the same as that on the respective control specimen. The schematic in figure 3 illustrates the test setup. Because of the low transparency and high absorptance of coating A, a gradient of 50 K was estimated to exist through its thickness at the maximum temperature of the thermal cycle. Therefore, to achieve a surface temperature of 1530 K, the control thermocouple for coating A was programed to reach 1480 K. Because of the high transparency and low absorptance of coating B, no gradient was assumed. Therefore, the control thermocouple for coating B was programed to reach 1530 K. Subsequent to the tests, data showed that A-coated specimens were overheated during some of the test cycles. The results of this overheating are discussed in the section entitled "Coating Integrity."

The thermoelectric properties of type R thermocouples can be affected by contact with carbon, silicon, and/or silica at high temperatures. Therefore, the control thermocouple from specimen 3 (coating A) was recalibrated after the thermal tests. No significant changes were observed in the thermoelectric properties of this thermocouple.

After each thermal cycle, specimens 1, 2, 4, and 5 were visually inspected for cracks and photographed as necessary to document degradation. After every fifth cycle, the test face of each specimen was examined with an X-ray diffractometer to monitor the amount of cristobalite on the coating surface. All X-ray diffraction examinations of the coating were conducted by scanning over the range $10^\circ \leq 2\theta \leq 30^\circ$ to observe the intensity of (101) reflections from cristobalite. A nickel-filtered $\text{CuK}\alpha$ source was used with a 3° beam slit and 0.02° detector slit. Calculations (ref. 2) showed that 90 percent of the intensity from diffracted X-rays was from the first 0.016 mm of coating thickness.

Testing at reduced pressure.— Specimen 7 (coated with LI-0042 as supplied by LMSC) was heated in a vacuum furnace (0.133 mPa) for 17 hr at 1530 K. The 17 hr was equivalent to the total time spent per specimen at 1530 K during the 100-cycle tests previously described. Continuous heating was used because of the complexity of conducting a cyclic test in the existing vacuum furnace and to insure more accurate temperature control. Before and after this exposure, the coating surface was examined with an X-ray diffractometer to determine the amount of cristobalite on the coating surface. After exposure, the specimen was visually inspected for signs of coating degradation.

Posttest Coating Examinations

After thermal cycling was completed, coating samples were taken from specimens 1, 2, 4, and 5 for powder diffraction analysis. The coating was pried loose from the specimens with a small metal spatula, and the back sides of coating fragments were scraped with a razor blade to remove adhering RSI. The coating samples were ground and passed through a 200-mesh sieve. Weight fractions of cristobalite in the powder samples were then determined with a standard quantitative X-ray diffraction technique.

Additional coating samples with adhering RSI not removed were taken from specimens for microscopic examination (optical and scanning-electron microscopy) and to determine the distribution of cristobalite through the coating thickness. Samples were mounted in epoxy to determine cristobalite distribution. The distribution was then determined by alternately scanning the coating surface in the X-ray diffractometer and removing fractions of the coating thickness. The X-ray techniques used were the same as those described in the section entitled "Thermal Testing." Coating was removed by wet grinding with No. 400 grit SiC paper. Grinding residue was removed by ultrasonic cleaning.

RESULTS

Photographs of specimens 1, 2, 4, and 5 taken after the 100-cycle tests are shown in figure 4. The cracks in coating A (specimens 1 and 2) were first observed after about 30 cycles (table II). The coating segments defined by the cracks were on the verge of spalling from the RSI. This was noted when the coatings were removed from the RSI for posttest analyses. Even though the coating was approaching spallation, removal of sizable segments (6 mm × 6 mm) was difficult; they tended to crumble because of the friability of the coating. In fact, the coating from specimen 1 crumbled so badly that a segment of sufficient size could not be obtained for determining cristobalite distribution through the coating thickness. No cracking, spalling, or other apparent degradation of the coating was observed on specimens 4 and 5.

Specimen shrinkages are reported in table II as the difference between specimen height at the center of the test face before and after testing. Specimens 1 and 2 (coating A) experienced about 4 times as much shrinkage as specimens 4 and 5 (coating B), respectively. Exposure to water also appeared to affect shrinkage, with the wet-tested specimens experiencing about twice as much as the dry.

Scanning-electron micrographs of coating cross sections from specimens 2 and 4 are shown in figure 5. These micrographs show how SiC affected the density of the coating. The borosilicate layer of coating A, which contained SiC, was quite porous when compared with that of coating B. Microscopic examination of the coating surfaces also revealed numerous open blisters on the surface of coating A. Although these cross sections were taken after 100 thermal exposures, the appearance of coating A is typical of "as-received" material except that coating porosity may have been slightly greater after thermal cycling.

The relative X-ray diffraction intensities of surface cristobalite on specimens 1, 2, 4, and 5 are plotted in figure 6 as a function of the number of thermal cycles. These data show that coating A (containing SiC) experienced much higher initial rates of devitrification and more total cristobalite formation than coating B (no SiC). After about 40 cycles, the cristobalite level in the surface of coating A remained relatively constant. The result of the 17-hr vacuum heating test (0.133 mPa at 1530 K) on specimen 7 is also shown in figure 6. This result is plotted at 100 cycles because 17 hr is roughly equivalent to the time that specimens 1, 2, 4, and 5 spent at 1530 K during 100 thermal cycles. Surface cristobalite level on specimen 7 was approximately equal to that of the dry coating B after 100 cycles.

Figure 7 shows the results of the quantitative X-ray diffraction analysis of the coating from specimens 1, 2, 4, and 5 after 100 thermal cycles. These data show that the total amount of cristobalite in coating A (SiC) was 3 to 5 times greater than that in coating B (no SiC).

The relative amount of cristobalite through the coating thickness is plotted in figure 8 for specimens 2, 4, and 5. Data were not obtained for specimen 1 because of the extreme friability of the coating. The distribution in the two coating types contrasted sharply. In coating A (specimen 2), the greatest concentration of cristobalite was in the base layer whereas the only appreciable concentration in coating B (specimens 4 and 5) was at the surface.

DISCUSSION

The major points to be made in the following discussion are:

- (1) Only the coating with SiC cracked.
- (2) Cracking did not occur prior to attaining a relatively high level of cristobalite in the coating surface.
- (3) Oxidation of SiC in the coating was a major devitrification mechanism.

Coating Integrity

The near-spalled condition of coating A that was noted during preparations for posttest analyses was not observed at the time cracks were first noted (30 to 35 cycles). Hence, the cracks probably helped promote further degradation of the interface between the coating and RSI by acting as paths for migration of water and oxygen to the interface. Water vapor and oxygen are contaminants that are known to enhance heterogeneous nucleation of cristobalite in amorphous silica (refs. 3 and 4). The additional cristobalite formation in the interface and the subsequent breakdown (crumbling) of interface material during thermal cycles probably led to the observed condition of incipient spallation. The results shown in figure 8 support this hypothesis. The largest concentration of cristobalite in coating A (specimen 2) was in the base layer of the coating.

The difference in shrinkage of the A- and B-coated specimens (table II) suggests that the A-coated specimens became hotter than the B-coated specimens during tests. A source of experimental error in temperature control was found which supports this theory. Control-temperature records showed that the surfaces of the A-coated specimens were about 50 K hotter than B-coated specimens for many of the cycles. The error apparently resulted because of drift in (or inadvertent changing of) the power limit control during the tests of coating A. (The same temperature program was used for all cycled specimens, and a power limiter control was used to vary the maximum desired temperature.) The difference in temperature, however, did not significantly affect the rates of devitrification, as suggested by the results of the vacuum-heating test of specimen 7 (LMSC coating with SiC). The vacuum test temperature was the same as the test temperature of coating B, and results showed that the vacuum specimen and the B-coated specimens (no SiC) had approximately the same amount of cristobalite after testing (fig. 6).

Devitrification Mechanisms

The data presented in figures 6, 7, and 8 show that both the rate of devitrification and the total amount of cristobalite formed in the LI-0042 were strongly influenced by the SiC in the borosilicate layer of the coating. SiC-induced devitrification can occur by two processes: SiC particles can act as sites for heterogeneous nucleation of cristobalite (nucleation caused by contact with impurity substances), or cristobalite can be formed directly from the oxidation of SiC particles. The latter is hypothesized to have been the dominant process for formation of cristobalite in the borosilicate layer although both processes may have contributed to cristobalite formation in the base layer.

Borosilicate glasses are generally very resistant to devitrification. Consequently, the SiC in coating A would not be expected to significantly affect cristobalite formation. On the other hand, the oxidation of SiC at temperatures above 1470 K and at partial pressures of oxygen greater than 1 Pa produces silica (SiO_2) plus carbon monoxide (CO) and/or carbon dioxide (CO_2). The SiO_2 can take the form of cristobalite (refs. 5, 6, and 7), and the oxidation of SiC can therefore account for the observed higher level of cristobalite in coating A. The high porosity of this coating (fig. 5) provides additional support for the SiC oxidation hypothesis. The gaseous by-products of oxidation (CO and CO_2) would be expected to produce coating porosity as they tried to escape from the low-viscosity coating. The SiC oxidation hypothesis is further supported by the results of the vacuum thermal test on specimen 7 (LMSC coating with SiC). The low partial pressure of oxygen during this test would severely limit the oxidation of SiC. As expected, the amount of cristobalite formed on the surface of this specimen was comparable to coating B. The devitrification observed in this specimen was probably due to the basic heterogeneous nucleation of cristobalite.

Cristobalite Distribution

The distribution of cristobalite in the coatings also appeared to be dependent on the presence or absence of SiC (fig. 8). In coating B, the cristobalite was almost entirely limited to a thin surface layer whereas the cristobalite was distributed through the entire thickness of coating A. Cristobalite was limited to the surface of coating B, probably because of the low porosity of the borosilicate layer and the absence of cracking. These circumstances resulted in a high resistance to penetration by water and oxygen. In coating A, SiC was distributed throughout the borosilicate layer. The oxidation of SiC during firing and subsequent heating resulted in a porous borosilicate layer that could easily be penetrated by contaminants. Although the penetrating contaminant (water, for example) would have only a slight effect on the borosilicate layer, the effect of its migration through the borosilicate to the base layer would be significant. The base layer (high-purity silica) has less resistance to heterogeneous nucleation of cristobalite and, if brought into contact with a migrating contaminant, would be susceptible to devitrification. The situation is further aggravated by the fact that the base layer is in intimate contact with SiC at the borosilicate—base-layer interface. The SiC particles could act as nucleation sites for cristobalite growth when in contact with base-layer glass. The expected distribution, then, would be that shown in figure 8 for coating A (specimen 2).

Effects of Water

The data presented in figures 6 and 7 show that exposure to water prior to each thermal cycle affected cristobalite formation. For coating A, observed differences in cristobalite X-ray intensities on the surface of the wet- and dry-tested specimens are not considered significant. The progressively degrading condition of these coatings significantly increased the scatter in the X-ray intensity measurements. For the wet B-coated specimens, the initial, more rapid rate of devitrification is consistent with cristobalite nucleation because of contamination with water. Since similar cristobalite growth rates were observed for both the wet and dry B-coated specimens, contamination apparently did not affect the kinetics of cristobalite growth.

The total amount of cristobalite formed in the wet-tested A-coated specimens was significantly greater than in the dry-tested A-coated specimens. This behavior was expected because water could penetrate the cracked and porous coating A and cause a rapid and more complete devitrification of the silica base layer which is relatively susceptible to devitrification. The lack of a significant difference between the total amount of cristobalite formed in the wet- and dry-tested specimens of coating B was also expected since this coating was relatively impervious to moisture penetration.

CONCLUDING REMARKS

The results of this investigation strongly suggest that silicon carbide (SiC) promoted the formation of cristobalite in the LI-0042 coating. Devitrification was significantly less when the coating was formulated without SiC powder or when the coating was heated at 1530 K in an oxygen-deficient atmosphere. Testing at a low partial pressure of oxygen suggested that devitrification in the borosilicate layer of the coating was related to the oxidation of SiC rather than to the heterogeneous nucleation of cristobalite that was caused by SiC as an impurity particle.

The incipient spalling of the LI-0042 coating after thermal cycling was attributed to the high concentration of cristobalite found in the silica base layer of the coating. This devitrification was probably the result of contamination of the silica base layer with either SiC or water and air which migrated through cracks and pores in the borosilicate layer.

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September 25, 1978

APPENDIX

APPLICATION OF LI-0042 COATING TO LIGHTWEIGHT THERMAL INSULATION

This appendix briefly summarizes the process specifications developed in reference 8 for applying LI-0042 to low-density fibrous silica materials such as RSI.

Preparation of Protective Coating Materials

Formulation of the coating shall be as given in the following table:

Material	Parts by weight	Weight, percent
Glass - 96-percent silica (Corning 7913)	380	49
Glass - borosilicate (Corning 7740)	20	3
Silicon carbide (No. 1200 grit)	20	3
0.5-percent methyl cellulose solution ^a	350	45

^aPrepared by dissolving 1 g of methyl cellulose (USP powdered, 4000 cP) in 200 cm³ deionized water (1 megohm).

The coating is prepared by adding the two glasses, the methyl cellulose solution, and silicon carbide to a plastic bottle and is then dispersed with an air-driven mechanical mixer. The coating slurry is then rolled on a ball mill base for at least 16 hr prior to application.

Application of Coating

The coating is applied in the following steps:

(1) Apply an initial dense silica layer by brushing on the fused silica material¹ with a soft, lint-free brush to a thickness of 0.07 to 0.2 mm.

(2) Dry the specimen for 30 min in an oven heated at 395 K.

(3) Place specimen in a furnace heated at 1480 ± 30 K for 15 ± 1 min. Then cool to room temperature.

(4) Apply coating slurry to a thickness of 0.23 to 0.30 mm by spraying (use air pressure of 275 to 345 kPa). Coating shall be applied with 7 to 8 passes and allowed to air dry for a minimum of 10 min.

¹Fused silica slip containing not less than 82-percent solids. The solids shall contain no less than 99.6-percent silica. The grain size shall indicate no greater than 3-percent retention on a standard 325-mesh sieve. The pH shall be within the range of 5 to 7.

APPENDIX

- (5) Dry the specimen for 30 min in an oven heated at 395 K.
- (6) Transfer specimen to a furnace heated at 920 ± 110 K for 10 to 20 min.
- (7) Place the specimen in a furnace heated at 1645 ± 15 K for 15 ± 1 min.
- (8) Cool to room temperature.

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TABLE I.- SPECIMENS

Specimen	Coating	SiC in coating	Type of thermal test	Exposure to H ₂ O
1	A	Yes	100 cycles to 1530 K at 101.3 kPa ↓ 17 hr in vacuum at 1530 K	No
2	A	Yes		Yes
a3	A	Yes		No
4	B	No		No
5	B	No		Yes
a6	B	No		No
7	LMSC	Yes		No

^aThermal control specimens containing thermocouples.

TABLE II.- TEST RESULTS

Specimen	Cycles before cracking	Shrinkage, mm
1	35	0.66
2	25	1.44
4	>100	.18
5	>100	.33
7	(a)	----

^aNo cracks after 17 hr of continuous vacuum exposure at 1530 K.

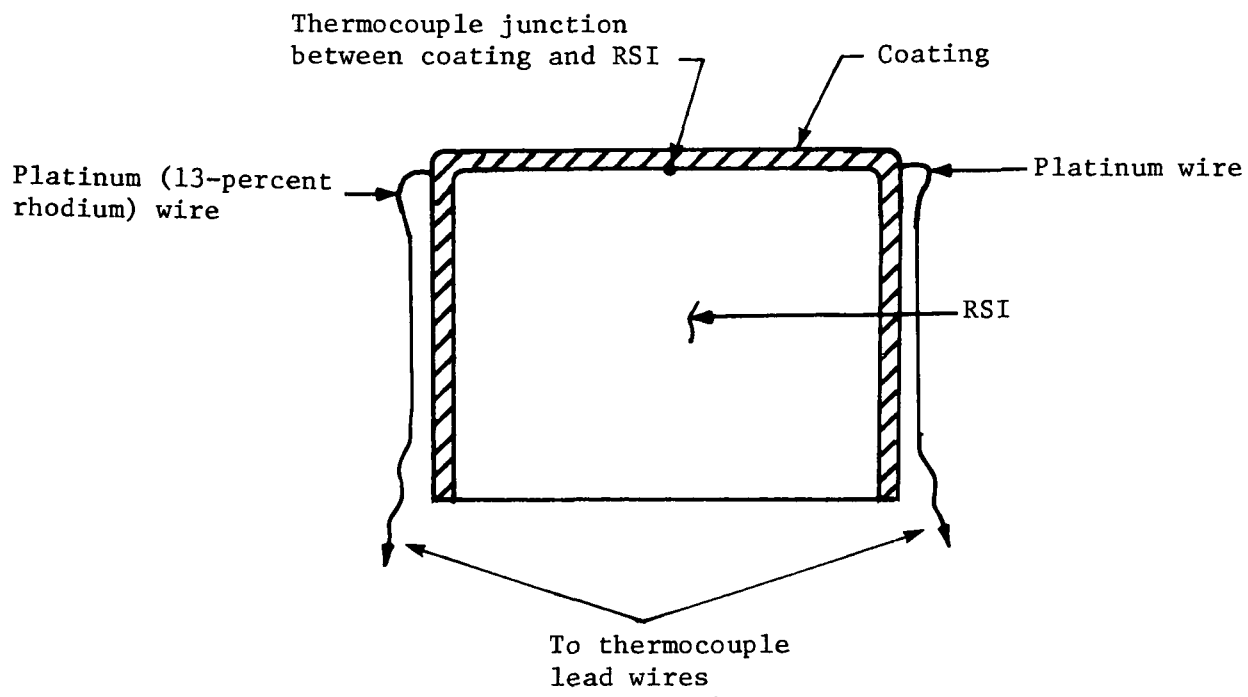


Figure 1.- Thermocouple installation in thermal control specimens.

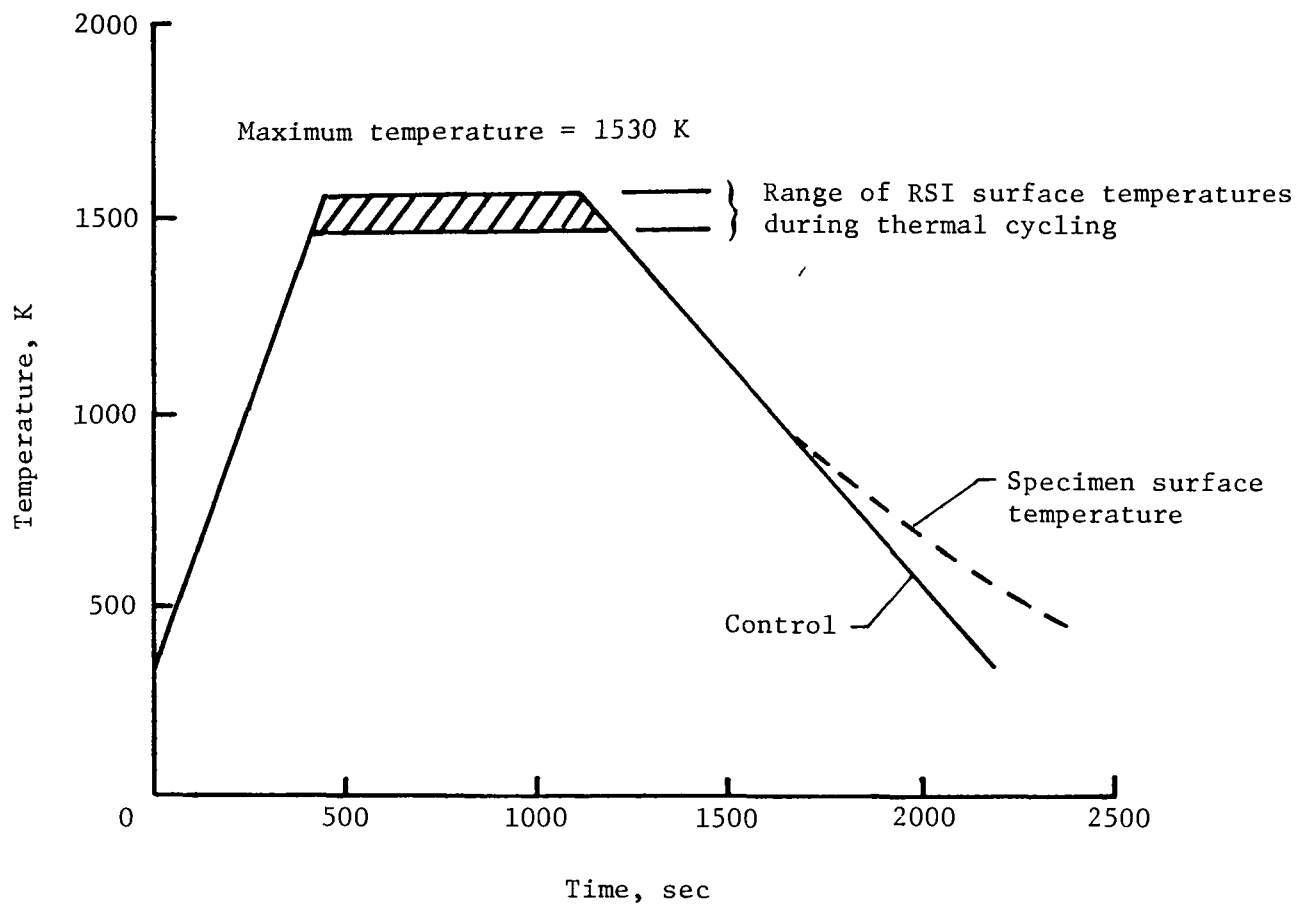


Figure 2.- Thermal cycle to simulate Space Shuttle reentry heating.

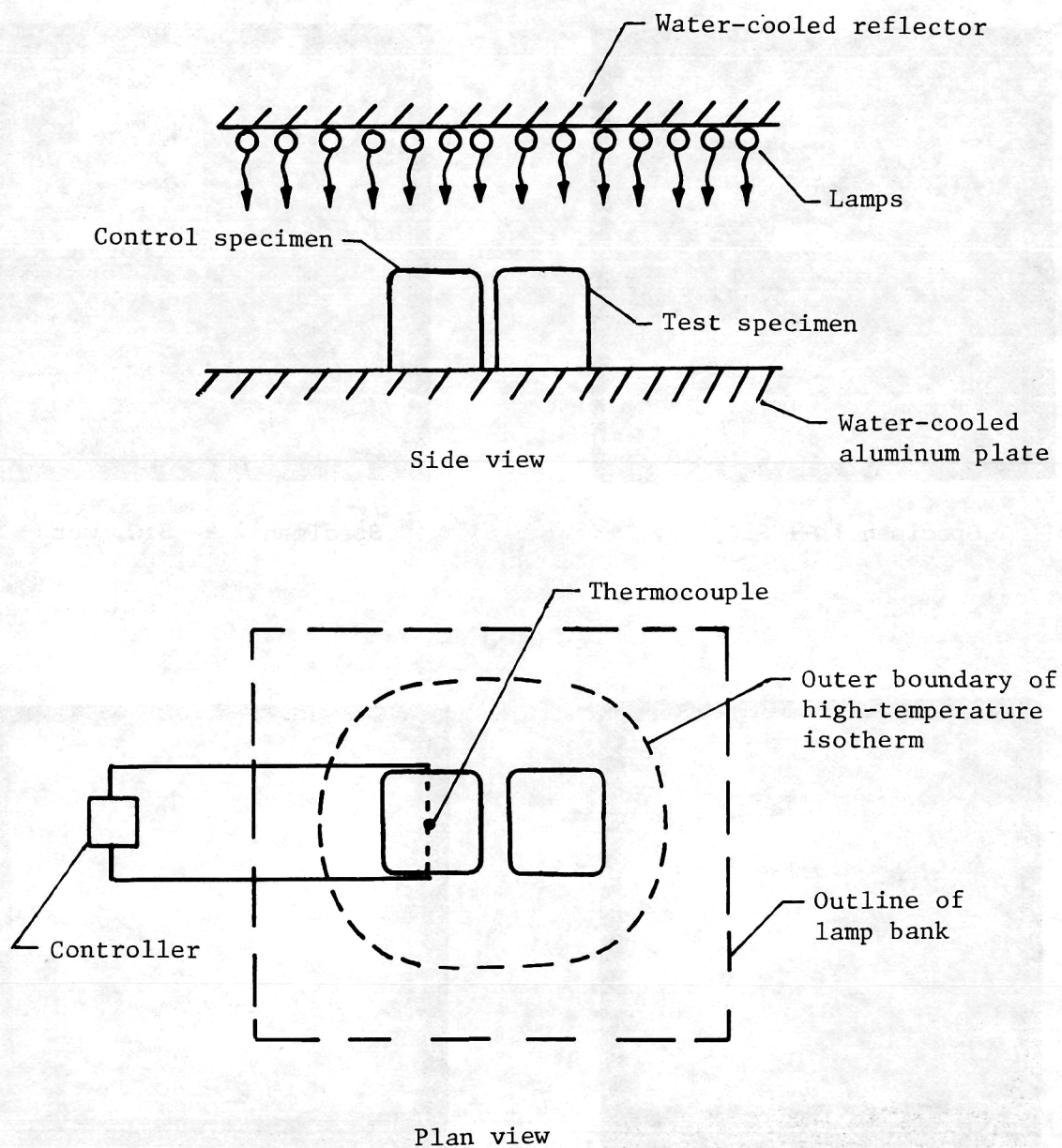
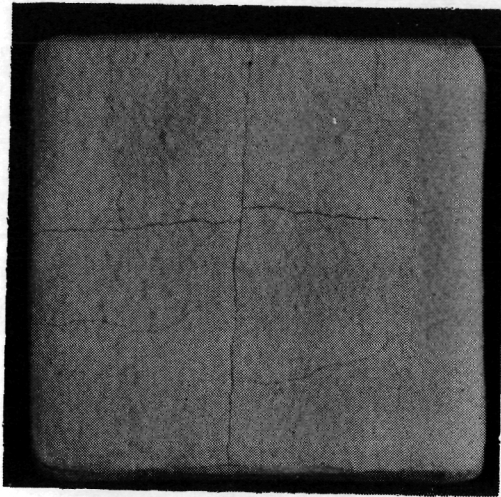
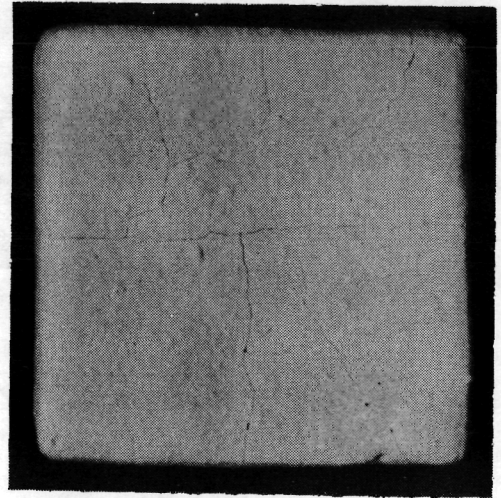


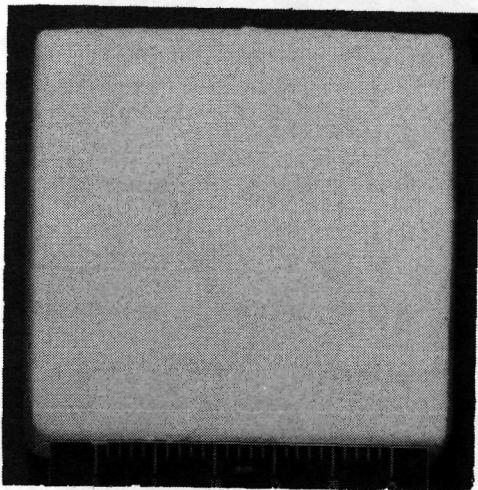
Figure 3.- Specimen orientation in radiant heater.



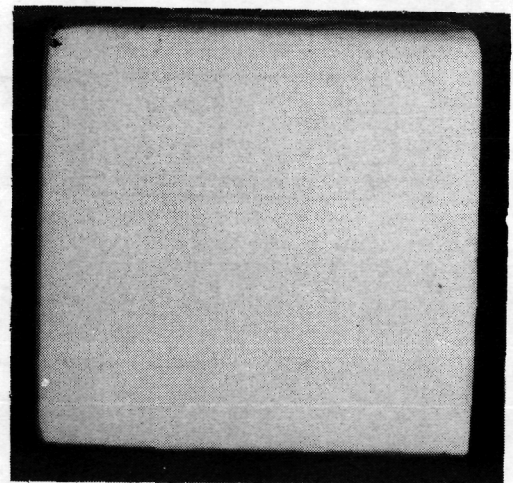
Specimen 1 — SiC, dry



Specimen 2 — SiC, wet

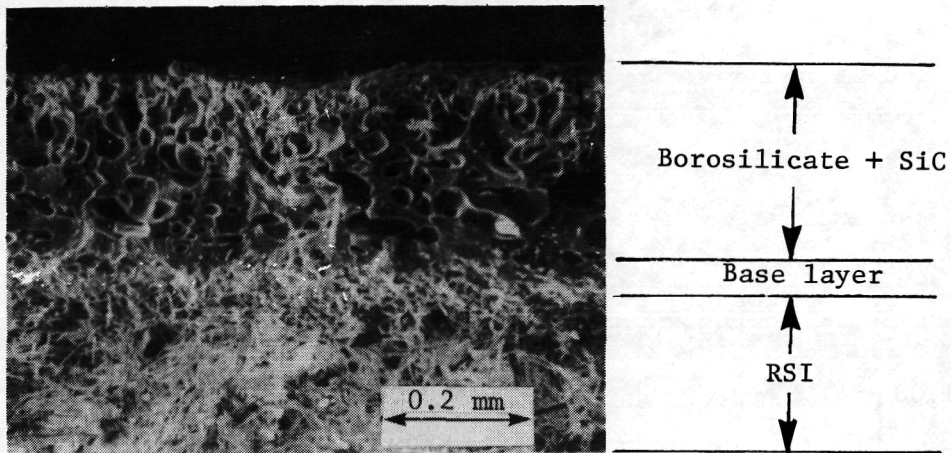


Specimen 4 — dry

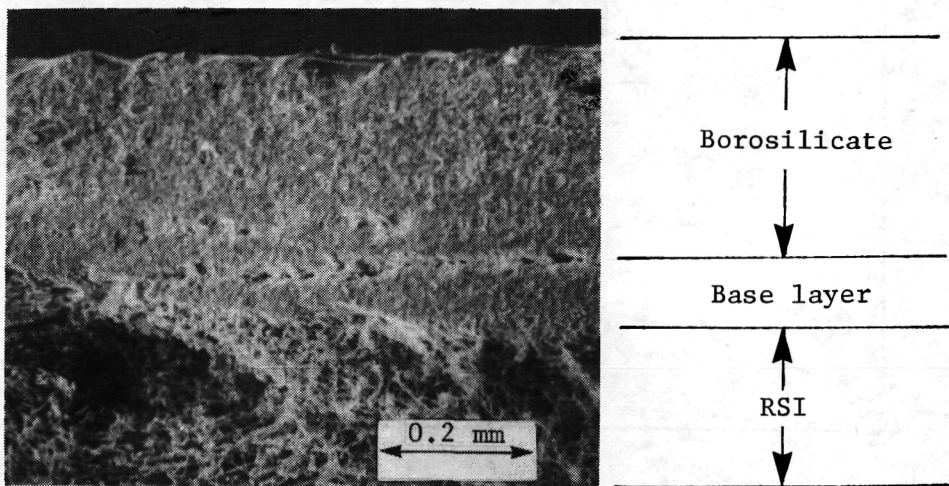


Specimen 5 — wet

Figure 4.- Specimens 1, 2, 4, and 5 after 100 thermal cycles. L-78-130



(a) LI-0042 with SiC at 100 thermal cycles.



(b) LI-0042 without SiC at 100 thermal cycles.

Figure 5.- Coating microstructure.

L-78-131

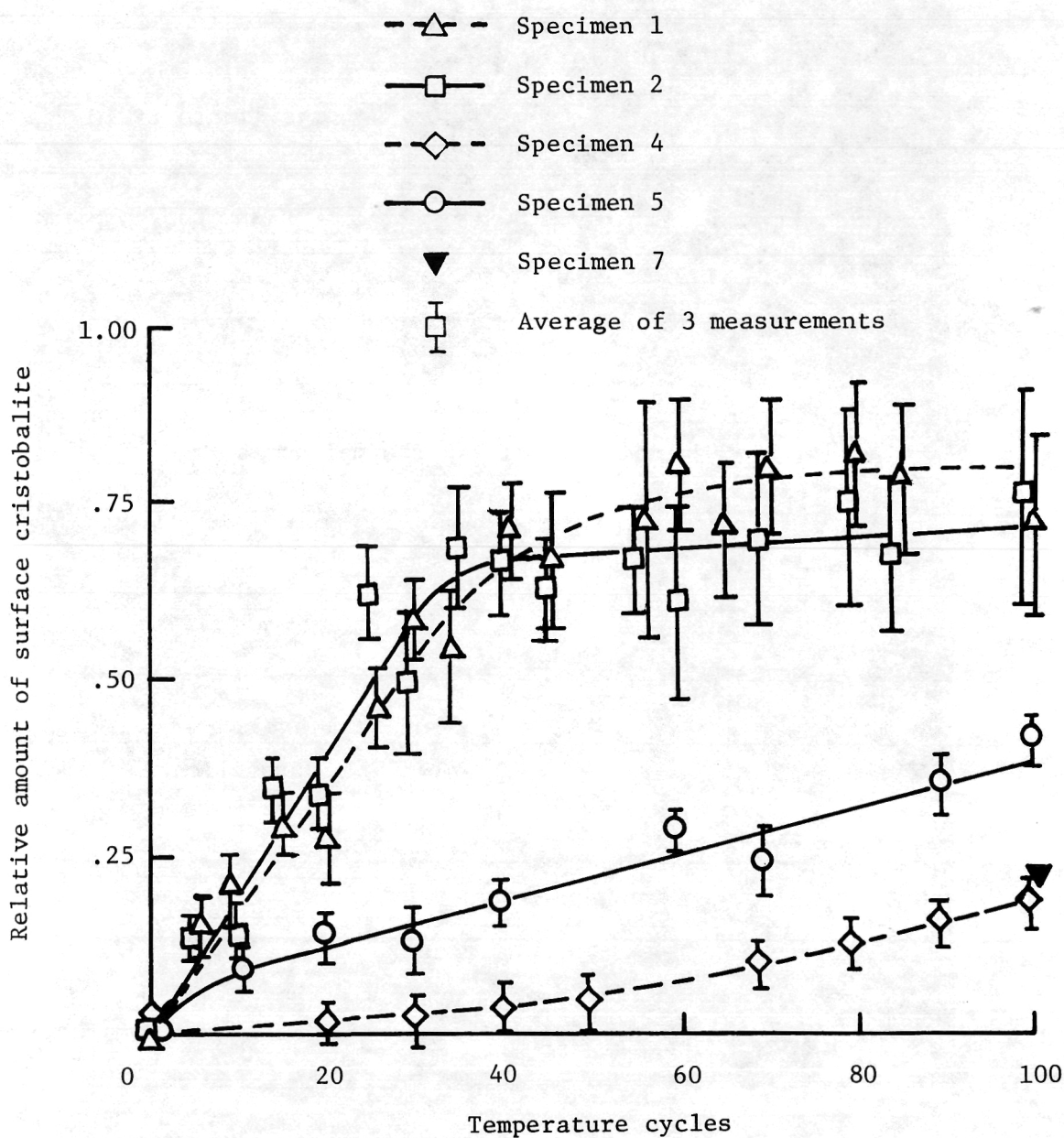


Figure 6.- The effect of thermal cycles on relative amounts of cristobalite on coating surfaces.

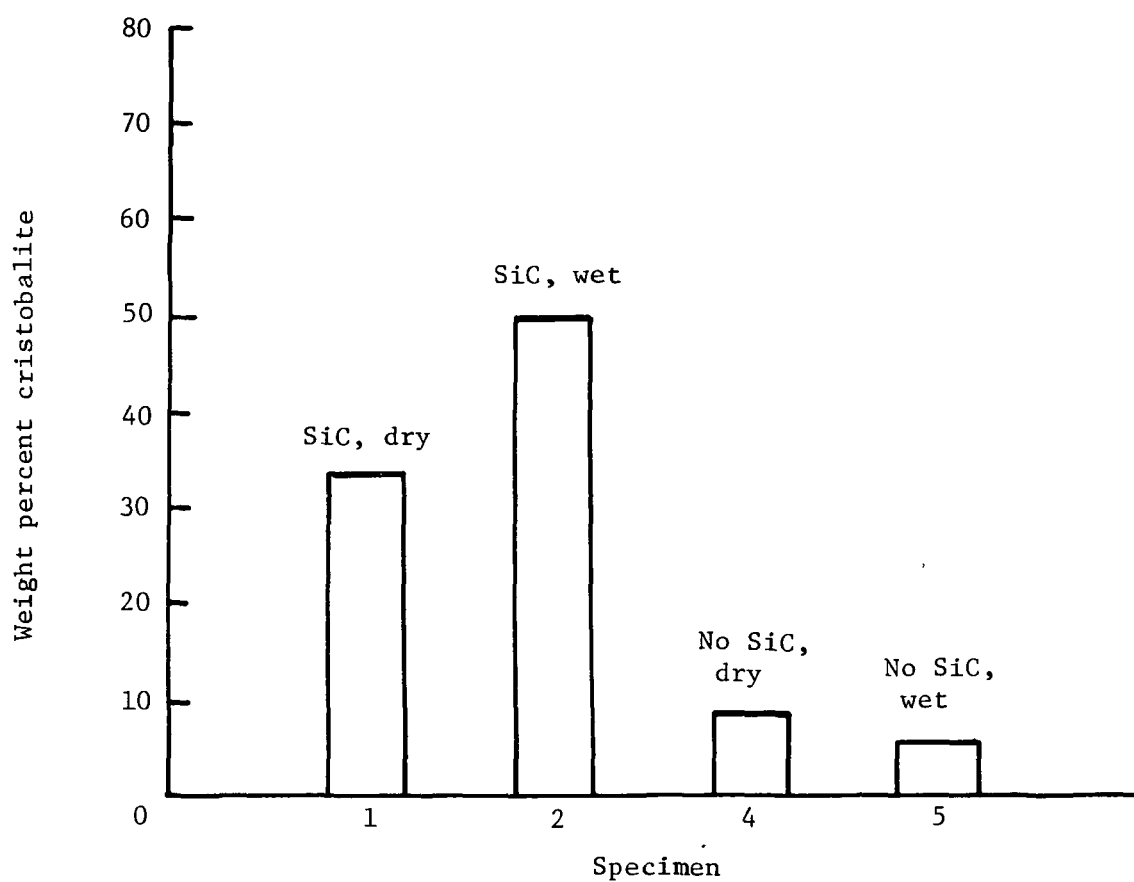


Figure 7.- Percent cristobalite in powdered coating samples after 100 thermal cycles.

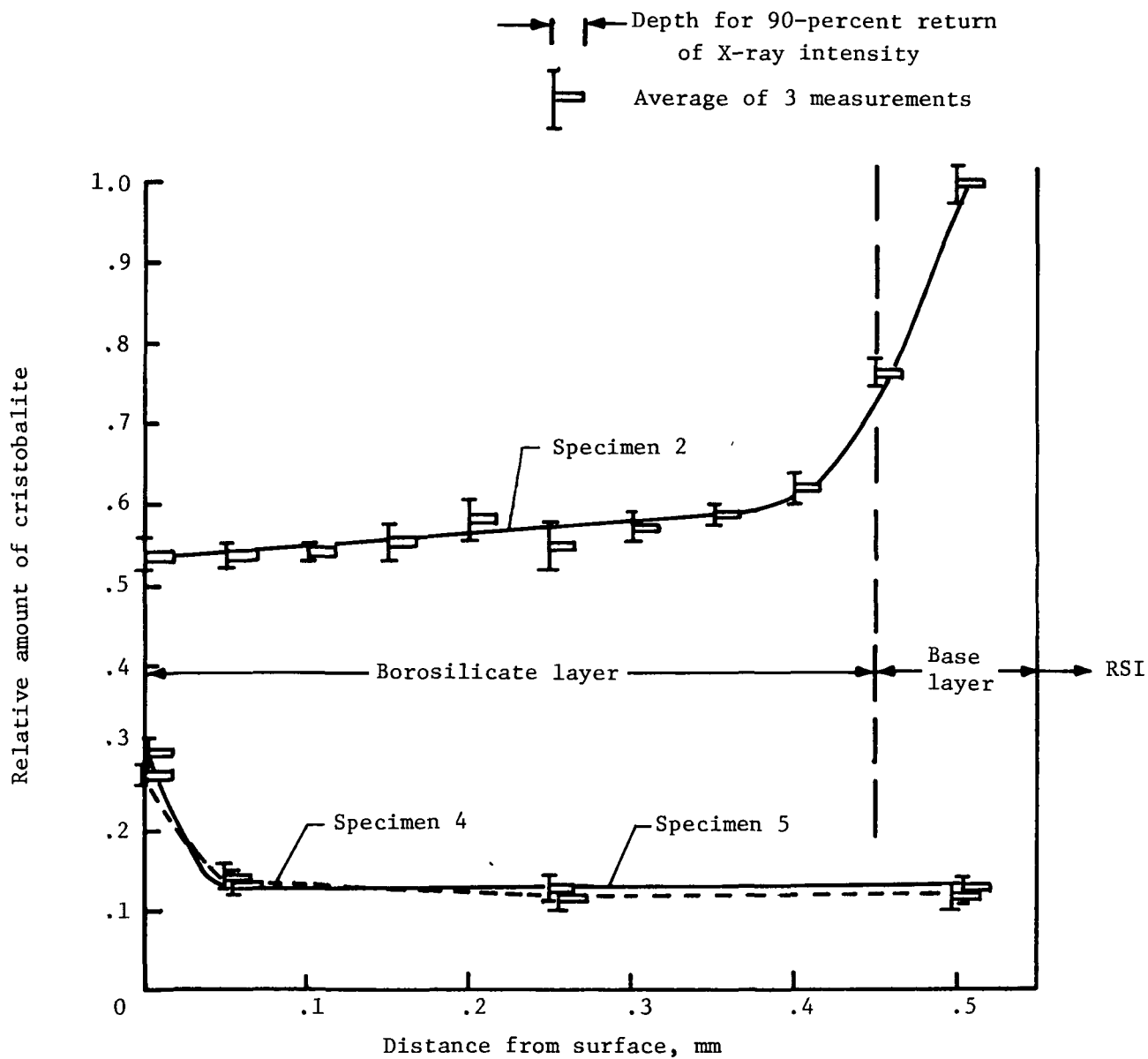


Figure 8.- Relative amounts of cristobalite throughout coating thickness.

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